Method of Test for CONSISTENCY OF EPOXY RESIN SYSTEMS

. DOTD Designation: TR 702-85 METHOD A

(Brookfield Viscosity - Mixed Systems)

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Scope

1. This method of test is intended to determine the consistency of mixed epoxy resin systems by measuring viscosity with the Brookfield viscometer.

Apparatus

- 2. (a) Balance A Type I or II, Class D balance conforming to AASHTO M 231.
- (b) Beaker A 400 ml graduated disposable plastic beaker.
- (c) Mixing Tools Stainless steel spatulas or wooden tongue depressors.
- (d) Viscometer Brookfield Viscometer, model RVT, with Model C helipath stand, or equivalent.
- (e) Spindle Brookfield No. 3 Cylindrical spindle, or equivalent.
- (f) Thermometer A thermometer conforming generally to the requirements for ASTM 1C or 1F thermometers.
- (g) Timer A clock or watch capable of measuring minutes and seconds.
- (h) Bath A temperature controlled water bath capable of maintaining 77 ± 2 $^{\rm O}$ F.

Safety Precautions

- 3. The following precautions should be observed when handling epoxy components and cleaning fluids:
- (a) Persons handling these materials should use appropriate protective clothing, including rubber or plastic gloves, and appropriate eye protection such as safety glasses.
- (b) If any epoxy or cleaning material should contact the skin, the material should be removed immediately with a dry cloth or paper towel, and the affected area should be washed thoroughly with soap and water.
- (c) If any material should come in contact with the eyes, flush immediately with water and contact a physician.
- (d) Adequate ventilation is necessary to prevent excessive inhalation of vapors.
- (e) Observe all precautions specified by the manufacturer before handling each material.

Sample Preparation

4. (a) Prior to mixing, condition the individual

components and any equipment with which they will come in contact, to the test temperature of 77 ± 2 °F by use of a water bath and/or laboratory temperature control.

- (b) Thoroughly stir the individual components, for at least one minute, immediately before testing.
- (c) Combine and mix sufficient quantities of components A and B, in accordance with the manufacturer's recommendations, such that a sample quantity of 400 ml is obtained. If the manufacturer does not recommend a mixing time, mix the sample for at least 3 minutes. Use separate mixing tools when obtaining and mixing the desired quantities of each component to avoid contamination. The sample should be visibly free of air voids resulting from mixing; if not, tap the beaker until the air voids are dispersed.

Procedure

- 5. (a) Ensure that the helipath stand is level. If leveling is required adjust the three set screws under the base of the stand until the bubble level is centered.
- (b) Attach the No. 3 cylindrical spindle to the lower shaft of the viscometer by screwing the spindle clockwise onto the shaft.
 - (c) Set the speed control knob to 20 rpm.
- (d) Ensure that the viscometer is level. If leveling is required, adjust the two set screws under the viscometer support bracket until viscometer bubble level is centered.
- (e) With the viscometer in the raised position, center the sample under the spindle.
- (f) Carefully lower the viscometer and spindle by use of the platform release lever taking care to avoid trapping air under the spindle plate until the spindle depth is at the immersion groove cut in the spindle's shaft.
- CAUTION: Care should be taken not to strike the spindle against the sides of the sample container since this could damage the shaft alignment.
- (g) Check to make sure the viscometer is still level. Make adjustments, if necessary, in accordance with paragraph 5 (d).
- (h) Depress the clutch and start the viscometer motor with the on/off switch and simultaneously start the stopwatch one minute after completion of any mixing. Release the clutch and allow the dial to rotate for 30 seconds.

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- (i) Depress the clutch and stop the instrument with the pointer in view. Record the reading obtained on the dial. Release clutch after reading is obtained.
- (j) Immediately after obtaining the readings, thoroughy clean the spindle and any spillage of the sample.

Calculations

6. Calculate the Brookfield viscosity according to the following formula:

 $V = R \times 0.5$

where:

V = Brookfield viscosity, in poises R = reading obtained on dial

Report

7. Report the Brookfield viscosity to the nearest poise.

Normal testing time is one day.

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METHOD B

(Brookfield Viscosity and Shear Ratio - Individual Components)

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Scope

1. This method of test is intended to determine the consistency and shear ratio of shear-rate-dependent epoxy components by measuring their viscosities with the Brookfield viscometer.

Apparatus

- 2. (a) Balance A Type I or II, Class D balance conforming to AASHTO M 231.
- (b) Beaker A 400 ml graduated disposable plastic beaker.
- (c) Mixing Tools Stainless steel spatulas or wooden tongue depressors.
- (d) Viscometer Brookfield Viscometer, model RVT, with Model C helipath stand, or equivalent.
- (e) Spindle T-Bar spindle letter T-D with chuck and spindle weight.
- (f) Thermometer A thermometer conforming generally to the requirements for ASTM 1C or 1F thermometers.
- (g) Timer A clock or watch capable of measuring minutes and seconds.
- (h) Bath A temperature controlled water bath capable of maintaining 77 ± 2 $^{\circ}$ F.

Safety Precautions

- 3. The following precautions should be observed when handling epoxy components and cleaning fluids:
- (a) Persons handling these materials should use appropriate protective clothing, including rubber or plastic gloves, and appropriate eye protection such as safety glasses.
- (b) If any epoxy or cleaning material should contact the skin, the material should be removed immediately with a dry cloth or paper towel, and the affected area should be washed thoroughly with soap and water.
- (c) If any material should come in contact with the eyes, flush immediately with water and contact a physician.
- (d) Adequate ventilation is necessary to prevent excessive inhalation of vapors.
- (e) Observe all precautions specified by the manufacturer before handling each material.

Sample Preparation

4. (a) Thoroughly stir the individual components

for at least one minute.

- (b) Pour 400 ml of each component into separate beakers.
- (c) Condition the components and any equipment with which they will come in contact to the test temperature of 77 ± 2 °F by use of a water bath and/or laboratory temperature control.
- (d) Mix the conditioned samples, for at least one minute, immediately before testing. Samples should be visibly free of air voids resulting from mixing; if not, tap the beaker until air voids are dispersed.

Procedure

- 5. (a) Ensure that the helipath stand is level. If leveling is required, adjust as described in paragraph 5(a) of Method A.
- (b) Attach the chuck to the viscometer shaft by screwing the upper chuck screw clockwise while holding the viscometer shaft stationary.

CAUTION: Care should be taken that the lower chuck screw is not tightened without spindle in place as it may damage the chuck.

- (c) Attach the spindle weight to the spindle chuck by turning the spindle weight clockwise while holding the lower chuck screw stationary.
- (d) Insert the spindle shaft into the weighted chuck and tighten the lower chuck screw while holding the viscometer shaft stationary.
 - (e) Set the speed control knob at 5.0 rpm.
- (f) Ensure that the viscometer is level. If leveling is required, adjust as described in paragraph 5(d) of Method A.
- (g) Center the sample under the spindle, with the viscometer in the raised position.
- (h) Carefully lower the viscometer and spindle by use of the platform release lever until the spindle bar touches the surface of the sample. Recheck the viscometer to ensure it is still level, and adjust if necessary in accordance with paragraph 5(d) of Method A.
- (i) Start the helipath stand and allow the spindle to penetrate into the sample until the spindle bar slightly penetrates the sample surface, at which time the helipath stand should be immediately stopped.
- (j) Simultaneously start stopwatch and helipath stand. Allow the spindle to penetrate the sample for 20 seconds to a depth of approximately 1/4 in. Stop the helipath stand and stopwatch immediately. The spindle is now at the proper starting test depth.

- (k) Depress the clutch and start the viscometer motor with the on/off switch. Release the clutch and allow the spindle to rotate at least one complete revolution.
- (1) Start the helipath stand and immediately record one reading per revolution of the dial for three consecutive readings. Take the reading at the same designated location on the dial glass of the viscometer for each of the three revolutions and record the average reading as (R).
- (m) Immediately clean the spindle and any spillage of epoxy before taking the next set of readings.
- (n) Repeat steps (f) through (m) to take readings at the 2.5 rpm and 0.5 rpm speeds. These readings shall be used to determine the shear ratio. If it is not possible to obtain the three readings in paragraph 5(1) due to operation at speeds such as 0.5 rpm, take as many readings as possible.

Calculations

6. (a) Calculate the Brookfield viscosity for each speed according to the following formula:

$$Vs = R \times f$$

where:

Vs = Brookfield viscosity, in poises, obtained at speed s

R = average reading obtained on the dial

f = factor from Table 1

(b) Calculate the shear ratio according to the following formula:

$$SR = \frac{V0.5}{V2.5}$$

where:

SR = shear ratio

V0.5 = viscosity at 0.5 rpm, poises

V2.5 = viscosity at 2.5 rpm, poises

Report

- 7. (a) Report the Brookfield viscosity to the nearest poise; the test temperature; and the test speed.
 - (b) Report the shear ratio to the nearest 0.1.

Normal testing time is one day.

TABLE I		
Spindle	Speed(s) rpm	$\frac{Factor(f)}{}$
T - D	5.0	40
T - D	2.5	80
T - D	0.5	400

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DOTD Designation: TR 702-85 METHOD C

(Sag Test - Mixed Systems)

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Scope

1. This method of test is intended to determine the consistency of mixed epoxy systems having high viscosities by measuring their sag characteristics.

Apparatus

- 2. (a) Balance A Type I or II, Class D balance conforming to AASHTO M 231.
- (b) Beaker A 250 ml (minimum) graduated disposable plastic beaker.
- (c) Template Metal template with a 3 by 6 in. rectangular opening. The thickness of the template shall be 0.10 ± 0.01 in.
- (d) Chart Paper Morest chart paper with a glazed finish on one side.
- (e) Mixing Tools Stainless steel spatulas or wooden tongue depressors.
- (f) 12-in. Ruler Graduated in 1/16 in. increments.
- (g) Thermometer A thermometer conforming generally to the requirements for ASTM 1C or 1F thermometers.
- (h) Bath A temperature controlled water bath capable of maintaining 77 ± 2 °F.
- (i) Timer A clock or watch capable of measuring minutes and seconds.

Safety Precautions

- 3. The following precautions should be observed when handling epoxy components and cleaning fluids:
- (a) Persons handling these materials should use appropriate protective clothing, including rubber or plastic gloves, and appropriate eye protection such as safety glasses.
- (b) If any epoxy or cleaning material should contact the skin, the material should be removed immediately with a dry cloth or paper towel, and the affected area should be washed thoroughly with soap and water.
- (c) If any material should come in contact with the eyes, flush immediately with water and contact a physician.
- (d) Adequate ventilation is necessary to prevent excessive inhalation of vapors.

(e) Observe all precautions specified by the manufacturer before handling each material.

Sample Preparation

- 4. (a) Prior to mixing, condition the individual components and any equipment with which they will come in contact, to the test temperature of 77 ± 2 °F by use of a water bath and/or laboratory temperature control.
- (b) Thoroughly stir the individual components, for at least one minute, immediately before testing.
- (c) Combine and mix sufficient quantities of components A and B, in accordance with the manufacturer's recommendations, such that a sample quantity of 200 ml is obtained. If the manufacturer does not recommend a mixing time, mix the sample for at least 3 minutes. Use separate mixing tools when obtaining and mixing the desired quantities of each component to avoid contamination.

Procedure

- 5. (a) Cut the chart paper approximately 1/4 inch greater than the exterior dimensions of the metal template.
- (b) Center the metal template on the chart paper. Adjust the template so that the 6 inch bottom edge of the template aligns with a horizontal block line on the chart paper.
- (c) Spread the mixed epoxy onto the glazed surface of the Morest chart inside the template to form a 3 by 6 by 0.10 ± 0.01 in. strip.
- (d) Carefully remove the metal template and clean as soon as possible.
- (e) Immediately support the chart vertically with the 6 in. strip in a horizontal position.
- (f) Allow the strip to cure for $24 \pm 1/2$ hours at 77 ± 2 °F.
- (g) Record the sag (the increase in width of the epoxy strip). This measurement shall be taken at the widest point after the 24 hour cure period.

Report

6. Report the sag to the nearest 1/16 in.

Normal testing time is one day.